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III. *An Analysis of a mineral Substance from North America, containing a Metal hitherto unknown.* By Charles Hatchett, Esq. F. R. S.

Read November 26, 1801.

IN the course of the last summer, when I was examining and arranging some minerals in the British Museum, I observed a small specimen of a dark-coloured heavy substance, which attracted my attention, on account of some resemblance which it had with the Siberian chromate of iron, on which at that time I was making experiments.

Upon referring to Sir HANS SLOANE's catalogue, I found that this specimen was only described as "a very heavy black stone, " with golden streaks," which proved to be yellow mica; and it appeared, that it had been sent, with various specimens of iron ores, to Sir HANS SLOANE, by Mr. WINTHROP, of Massachusetts. The name of the mine, or place where it was found, is also noted in the catalogue; the writing however is scarcely legible: it appears to be an Indian name, (Nautneauge;) but I am informed by several American gentlemen, that many of the Indian names (by which certain small districts, hills, &c. were forty or fifty years ago distinguished,) are now totally forgotten, and European names have been adopted in the room of them. This may have been the case in the present instance; but, as the other specimens sent by Mr. WINTHROP were from the mines of Massachusetts, there is every reason to believe that the

mineral substance in question came from one of them, although it may not now be easy to identify the particular mine.

§ I. DESCRIPTION OF THE ORE.

The external colour is dark brownish gray.

The internal colour is the same, inclining to iron gray.

The longitudinal fracture is imperfectly lamellated; and the cross fracture shews a fine grain.

The lustre is vitreous, slightly inclining in some parts to metallic lustre.

It is moderately hard, and is very brittle.

The colour of the streak or powder is dark chocolate brown.

The particles are not attracted by the magnet.

The specific gravity, at temp. 65°, is 5918.*

Experiment 1.

Some of the ore, reduced to fine powder, was digested in boiling muriatic acid for about one hour.

The acid appeared to have acted but slightly upon the powder; as the former remained colourless, and the latter did not seem to be diminished. A portion, however, chiefly of iron, was found to be dissolved; for ammonia formed a yellow flocculent precipitate; prussiate of potash produced one which was blue;

* The following results of some experiments which I have purposely made, will shew how much the specific gravity of this ore is different from that of Wolfram, and Siberian chromate of iron.

Pure Wolfram, free from extraneous substances, at temp. 65°	-	-	6955.
Siberian chromate of iron, containing some of the green oxide	-	-	3728.
Pure Siberian chromate of iron	-	-	4355.

The Siberian chromate of iron, like all other mineral substances which are not crystallized, and which consequently are not always homogeneous, must evidently be liable to considerable variations in specific gravity.

and tincture of galls, when the excess of acid had been previously saturated by an alkali, formed a precipitate of a rich purplish brown colour.

Experiment II.

Another portion of the powder was, in like manner, digested with nitric acid; but, excepting some slight traces of iron, this acid afforded nothing worthy of notice; the action of it upon the ore, was indeed scarcely perceptible.

Experiment III.

Some of the pulverized ore was digested with concentrated sulphuric acid, in a strongly-heated sand-bath, until nearly the whole of the acid was evaporated; the edges of the mass then appeared bluish, and became white, when boiling distilled water was added.

This acid certainly acted much more powerfully than those which have been mentioned; but still only a small part of the ore was dissolved. It must however be observed, that a very copious blue precipitate was obtained by prussiate of potash; a plentiful purplish brown precipitate was also produced by tincture of galls, after the excess of acid had been saturated by an alkali; and, lastly, when the yellow ferruginous precipitate formed by ammonia was dissolved in diluted nitric acid, some white flocculi remained, which were completely insoluble in the acid, even when it was added so as to be in considerable excess.

From these experiments it was evident, that the ore could not readily be decomposed by the direct application of the mineral acids; and I therefore had recourse to the following

method, which has frequently been employed with success in similar cases.

ANALYSIS.

A.

A mixture of 200 grains of the powdered ore with five times the weight of carbonate of potash, was exposed to a strong red heat, in a silver crucible. As soon as the matter began to flow, a very perceptible effervescence took place; and, when this had subsided, the whole was poured into a proper vessel.

The mass, when cold, was grayish-brown.

Boiling distilled water was poured upon it; and the brown residuum, which was considerable, was welledulcorated upon a filter.

The filtrated liquor had a slight yellowish tinge, and, being supersaturated with nitric acid, afforded a copious white flocculent precipitate, which speedily subsided; but, although a very considerable additional quantity of nitric acid was poured upon the precipitate, it was not re-dissolved.

The residuum of the ore was dark brown, and was again melted with potash, and treated as before; but scarcely any effect was thus produced; the alkali was therefore washed off, and the powder was digested with muriatic acid, which soon assumed the deep yellow colour usually communicated to it by iron. After half an hour, the acid was decanted, and the residuum was washed with distilled water.

This powder was now of a much paler colour; and, being mixed with potash, it was melted and treated as before. A considerable precipitate was again obtained by the addition of nitric acid; and the residuum, after being digested with muriatic acid, was again fused with potash, by which means the

whole was completely decomposed, after about five repetitions of each operation.

B.

The muriatic solution was diluted, and, being saturated with ammonia, afforded a plentiful ochraceous precipitate; which again was dissolved in cold dilute nitric acid, and afforded a small quantity of a white insoluble substance, similar to that which was obtained from the alkaline solution. From this nitric solution, I then obtained, by means of ammonia, a precipitate of oxide of iron, which, being properly dried, weighed 40 grains.

C.

The different alkaline solutions which had been made subsequent to that which has been first mentioned, were mixed together, and, being supersaturated with nitric acid, afforded the same white insoluble precipitate; the total quantity of which, obtained from 200 grains of the ore, amounted to about 155 grains.

The liquor from which this precipitate had been separated by nitric acid, was then saturated with ammonia, and, being boiled, afforded about 2 grains of oxide of iron.

I obtained, therefore, from 200 grains of the ore,

	Grains.	
Oxide of iron	- - -	42
And of the white precipitated substance		155
		} = 197.

But, as I could not repeat the analysis without destroying the remaining part of the only specimen at present known of this ore, I do not wish the above stated proportions to be regarded as rigidly exact; it will be sufficient, therefore, to say at present, that the ore is composed of about three parts of the white matter, and rather less than one of iron.

§ II. PROPERTIES OF THE WHITE PRECIPITATE.

A.

It is of a pure white, and is not extremely heavy.

It has scarcely any perceptible flavour, nor does it appear to be soluble in boiling water; when, however, some of the powder is placed upon litmus paper moistened with distilled water, the paper in a few minutes evidently becomes red.

B.

1. When examined by the blow-pipe, it is not fusible *per se* in a spoon of platina, nor upon charcoal, but only becomes of a less brilliant white.

2. Borax does not appear to act upon it; for the white particles are only dispersed throughout the globule.

3. It produces an effervescence when fused with carbonate of soda, and forms a colourless salt; but, if too much of it be added, then the mass, when cold, appears like a white opaque enamel.

4. When carbonate of potash is employed, the effects are similar in every respect to those of soda; and it may here be remarked, that the saline combinations thus formed with soda, or potash, are soluble in water; and that these solutions have the same properties as that which was formed when the ore was decomposed by an alkali. The portion of the white precipitate which may be in excess, subsides unaltered, when the globules are dissolved in water.

5. Phosphate of ammonia produces a very marked effect; for, when melted in a platina spoon, if some of the white substance be added, a considerable effervescence takes place, and the two substances rapidly unite. The globule, when cold, is

deep blue, with a tinge of purple, but, when held between the eye and the light, it appears of a greenish gray colour.

C.

It is perfectly insoluble, and remains unchanged in colour, and in every other respect, when digested in boiling concentrated nitric acid.

D.

It is dissolved by boiling sulphuric acid, and forms a transparent colourless solution, which is however only permanent while the acid remains in a concentrated state; for, if a large quantity of water be added to the solution, or if the latter be poured into a vessel of distilled water, the whole in a few minutes assumes a milky appearance, and a white precipitate is gradually deposited, which cracks as it becomes dry upon the filter, and, from white, changes to a lavender-blue colour, and again, when completely dry, to a brownish gray. It is then insoluble in water, has not any flavour, is semi-transparent, and breaks with a glossy vitreous fracture.

This substance is much heavier than the original white precipitate; and in a very slight degree may be dissolved by boiling muriatic acid, or by boiling lixivium of potash.

Upon examining these solutions, I found that both contained the original white substance, together with some sulphuric acid; so that the precipitate obtained from the sulphuric solution by the addition of water, is a sulphate of the white matter.*

The whole is not however precipitated by water; for a part

* This sulphate is also precipitated when the sulphuric solution has been long exposed in an open vessel to the air; and, according as this may be moist or dry, the effect is produced sooner or later.

remains in solution, which may be separated from the sulphuric acid by either of the fixed alkalis, or by ammonia.

The sulphuric solution is not rendered turbid by the addition of water, until some minutes at least have elapsed; when, therefore, some prussiate of potash was added immediately after the water, the colour of the liquor became olive green, and a copious precipitate, of a beautiful olive colour, was gradually deposited.

Tincture of galls, after a few minutes, caused the liquor to become turbid, and a very high orange-coloured precipitate was obtained.

A few drops of phosphoric acid were added to a part of the concentrated sulphuric solution; and, after about 12 hours, the whole became a white opaque stiff jelly, which was insoluble in water.

Potash, soda, and ammonia, whether pure or in the state of carbonates, separate the substance in question from the sulphuric solution, in the form of a white flocculent precipitate; and, when these alkalis are added to a considerable excess, they do not redissolve the precipitate, unless they are heated; then, indeed, the fixed alkalis act upon it, and form combinations which have already been mentioned, but which we shall soon have occasion more particularly to notice.

E.

1. The white precipitate, when recently separated from potash, is soluble in boiling muriatic acid; and this solution may be considerably diluted with water, without any change being produced.

2. A part was evaporated to dryness, and left a pale yellow substance, which was not soluble in water, and was dissolved

with great difficulty, when it was again digested with muriatic acid.

3. Prussiate of potash changed the colour of the muriatic solution to an olive-green; the liquor then gradually became turbid, and an olive-coloured precipitate was obtained, similar to that which has been lately mentioned. But,

4. If some nitric acid was previously added to the muriatic solution, then the prussiate changed the liquor to a grass-green, but did not produce any precipitate.

5. Tincture of galls, in a few minutes, formed an orange-coloured precipitate, like that which has been mentioned; but, if the acid was in too great an excess, it was necessary to add a small quantity of lixivium of potash or soda, before the precipitate could be obtained.

6. A small quantity of phosphoric acid, being added to the muriatic solution, in a few hours formed a white flocculent precipitate.

7. Potash, soda, and ammonia, also produced white flocculent precipitates, which were not redissolved by an excess of the alkalis, unless the liquors were heated; and, in that case, part was dissolved by the fixed alkalis, but not by ammonia.

8. The muriatic solution did not yield any precipitate, when the muriates of lime, magnesia, and strontian, were added; but muriate of barytes formed a slight cloud.

9. When a piece of zinc was immersed in the muriatic solution, a white flocculent precipitate was obtained.*

* This appears to indicate the obstinacy with which this substance retains a certain portion of oxygen; for we here see that zinc does not precipitate it in the metallic state, but only reduces it to an insoluble oxide.

F.

The acetous acid has not any apparent effect on the white precipitate, when long digested with it.

G.

The fixed alkalis readily combine with this substance, both in the dry and in the humid way.

We have already seen, that the former method was employed with success in the analysis of the ore; and the experiments made with the blow-pipe may be regarded as an additional confirmation. In each of these cases, the white precipitate combined with the alkali, as soon as the heat was sufficient to cause the latter to flow; and, when a carbonate was employed, a portion of carbonic acid was expelled.

The carbonic acid was in like manner disengaged, when the white precipitate was boiled with lixivium of carbonate of potash, or of soda; and the solutions thus prepared, resembled in every respect those which were formed by dissolving in water the salts which had been produced in the dry way.

It will be proper here to give a more particular account of these combinations.

1. Some of the white precipitate was digested, during nearly one hour, with boiling lixivium of pure or caustic potash: about one-fourth of the powder was dissolved; and the remainder, which appeared little if at all altered, subsided to the bottom of the vessel.

The clear solution, which contained a great excess of alkali, was decanted; and, by gentle evaporation, yielded a white glittering salt, in scales, very much resembling the concrete boracic acid.

The salt was placed upon a filter, so that the lixivium might be separated. It was then washed with a small quantity of cold distilled water; and, being dried, remained as above described, although constantly exposed to the open air.

This salt had an acrid disagreeable flavour, and contained a small excess of alkali. It did not dissolve very readily in cold water; but, when dissolved, the solution was perfect and permanent.

Some nitric acid was added to part of the solution, and immediately rendered it white and turbid. In a short time, a white precipitate was collected, similar to that which had been employed to neutralise the potash; and the clear supernatant liquor, being evaporated, only afforded nitre.

Prussiate of potash was added to another portion; but did not produce any effect, until some muriatic acid was dropped into the liquor, which then immediately assumed a tinge of olive green, and slowly deposited a precipitate of the same colour.

Tincture of galls did not affect the solution at first; but, when a few drops of muriatic acid had been added, it gradually lost its transparency, and yielded an orange-coloured precipitate.

2. As so large a part of the white precipitate had remained undissolved in the foregoing experiment, it was digested again with another portion of the same lixivium, but without any effect. I therefore washed off the alkali, and boiled some nitric acid with the powder, until the acid was completely evaporated. After this, the powder was exposed to a strong heat in a sand-bath. It was then again digested with the lixivium, and a part was dissolved as before; but still the residuum required to be treated with nitric acid, before the alkaline liquor could again act upon it; so that it was necessary to repeat these alternate

operations several times, before the whole of the powder could be united with the alkali.

3. When the white precipitate was digested with solution of carbonate of potash, or of soda, it was dissolved, much in the same manner as above related; and the properties of the solutions, when examined by reagents, were also similar, excepting that the orange-coloured precipitates produced by tincture of galls were of a paler colour.

Tungstate of potash, molybdate of potash, and cobaltate of ammonia, being severally added to the solution of the white substance in potash, produced white flocculent precipitates.

Hydro-sulphuret of ammonia produced a reddish chocolate-coloured precipitate.

4. As the ore was decomposed by being fused with potash, the following experiment affords a curious instance (among the many already known) of the change in the order of affinities produced by a difference of temperature.

Some of the solution of the white precipitate in potash, was poured into the alkaline solution of iron, which was formerly known by the name of STAHL'S *Tinctura Alkalina Martis*. Potash was in excess in both of these solutions; but nevertheless a cloud was immediately produced, and a brown ferruginous precipitate was deposited.

Part of this precipitate was dissolved in muriatic acid; and the solution, being examined in the usual way, yielded a blue precipitate when prussiate of potash was added, and a purplish brown precipitate with tincture of galls.

The other part of the precipitate was digested with dilute nitric acid; which dissolved the ferruginous part, but left untouched a white flocculent matter, perfectly resembling the

substance which has been so often mentioned. The precipitate therefore produced by the mixture of the two alkaline solutions, was a combination of the white matter with oxide of iron, very similar to the original ore.

H.

The white precipitate, when distilled with four parts of sulphur, remained pulverulent, and, from white, was only changed to a pale ash colour.

Nitric acid was digested on the powder, and, being heated, afforded some nitrous gas ; after this, the powder became white, and in every respect recovered its original properties.

I.

Before I conclude this section, I must observe, that when the olive-green precipitates, obtained by prussiate of potash, were digested in an alkaline lixivium, they were decomposed ; for the alkali combined with the prussic acid, and with a small part of the white matter ; but the greater part of the latter remained undissolved, in the same white flocculent state which was noticed when the alkaline combinations were mentioned.

The orange-coloured precipitates, formed by tincture of galls, were also decomposed when digested in boiling nitric acid ; and the white matter was recovered in its original state.

§ III. REMARKS.

The preceding experiments shew, that the ore which has been analysed, consists of iron combined with an unknown substance, and that the latter constitutes more than three-fourths of the whole. This substance is proved to be of a metallic nature, by the coloured precipitates which it forms with prussiate of potash, and with tincture of galls ; by the effects which zinc

produces, when immersed in the acid solutions; and by the colour which it communicates to phosphate of ammonia, or rather to concrete phosphoric acid, when melted with it.

Moreover, from the experiments made with the blow-pipe, it seems to be one of those metallic substances which retain oxygen with great obstinacy, and are therefore of difficult reduction.

It is an acidifiable metal; for the oxide reddens litmus paper, expels carbonic acid, and forms combinations with the fixed alkalis. But it is very different from the acidifiable metals which have of late been discovered; for,

1. It remains white when digested with nitric acid.
2. It is soluble in the sulphuric and muriatic acids, and forms colourless solutions, from which it may be precipitated, in the state of a white flocculent oxide, by zinc, by the fixed alkalis, and by ammonia. Water also precipitates it from the sulphuric solution, in the state of a sulphate.
3. Prussiate of potash produces a copious and beautiful olive-green precipitate.
4. Tincture of galls forms orange or deep yellow precipitates.
5. Unlike the other metallic acids, it refuses to unite with ammonia.
6. When mixed and distilled with sulphur, it does not combine with it so as to form a metallic sulphuret.
7. It does not tinge any of the fluxes, except phosphoric acid, with which, even in the humid way, it appears to have a very great affinity.
8. When combined with potash and dissolved in water, it forms precipitates, upon being added to solutions of tungstate of potash, molybdate of potash, cobaltate of ammonia, and the alkaline solution of iron.

These properties completely distinguish it from the other

acidifiable metals, *viz.* arsenic, tungsten, molybdena, and chromium; as to the other metals lately discovered, such as uranium, titanium, and tellurium, they are still farther removed from it.

The colours of the precipitates produced by prussiate of potash and tincture of galls, approach the nearest to those afforded by titanium. But the prussiate of the latter is much browner; and the gallate is not of an orange colour, but of a brownish red, inclining to the colour of blood. Besides, even if these precipitates were more like each other, still the obstinacy with which titanium refuses to unite with the fixed alkalis, and the insolubility of it in acids when heated, sufficiently denote the different nature of these two substances.

The iron in the ore which has been examined, is apparently in the same state as it is in wolfram, *viz.* brown oxide; and this oxide is mineralised by the metallic acid which has been described, in the same manner as the oxides of iron and manganese are mineralised by the tungstic acid or rather oxide. For, from several experiments made upon a large scale, I have reason to believe that in wolfram, the tungsten has not attained the maximum of oxidation. Several facts in the course of the experiments lately described, seem to prove, that this new metal differs from tungsten and the other acidifiable metals, by a more limited extent of oxidation; for, unlike these, it seems to be incapable of retaining oxygen sufficient to enable the total quantity to combine with the fixed alkalis. In § II. G. 2, this is very evident; for, from the experiment there described it appears, that when the metallic acid or oxide was digested with lixivium of potash, only a part was dissolved; and that the remainder was insoluble in the same lixivium, till it had received

an additional portion of oxygen, by being treated with nitric acid; also that several of these alternate operations were required, before any given quantity of the metallic oxide could be completely combined with the alkali. Now there is much reason to believe, that in this case, when the metallic oxide or acid was digested with potash, the portion which was dissolved, received an accession of oxygen at the expense of the other part, which of course was thus reduced to the state of an insoluble oxide, and therefore required to be again oxidated by nitric acid, before it could combine with the alkaline solution; but still it appeared, that an adequate proportion of oxygen could never be superinduced, so as to render the oxide totally and immediately soluble in the alkalis by one operation, or even by two.

We may, therefore, regard this as an instance of the effects resulting from disposing affinity, and as very similar to those observed in respect to copper, which have been noticed by my ingenious friend Mr. CHENEVIX, in his valuable analysis of the arseniates of copper and of iron.*

My researches into the properties of this metal, have of course been much limited by the smallness of the quantity which I had to operate upon; but I flatter myself that more of the ore may soon be procured from the Massachuset mines, particularly as a gentleman now in England, (Mr. SMITH, Secretary to the American Philosophical Society,) has obligingly offered his assistance on this occasion. We shall then be able more fully to investigate the nature of this substance; and shall be more capable of judging how far it may be applicable to useful purposes. At present, all that can be said is, that the olive green prussiate and the orange-coloured gallate are fine colours;

* Phil. Trans. for 1801, p. 233.

and, as they do not appear to fade when exposed to light and air, they might probably be employed with advantage as pigments.

I am much inclined to believe, that the time is perhaps not very distant, when some of the newly-discovered metals, and other substances, which are now considered as simple, primitive, and distinct bodies, will be found to be compounds. Yet I only entertain and state this opinion as a probability; for, until an advanced state of chemical knowledge shall enable us to compose, or at least to decompose, these bodies, each must be classed and denominated as a substance *sui generis*. Considering, therefore, that the metal which has been examined is so very different from those hitherto discovered, it appeared proper that it should be distinguished by a peculiar name; and, having consulted with several of the eminent and ingenious chemists of this country, I have been induced to give it the name of Columbium.

POSTSCRIPT.

It appears proper to mention some unsuccessful attempts, which I have lately made to reduce the white oxide.

Fifty grains were put into a crucible coated with charcoal; and, being covered with the same, the crucible was closely luted, and was exposed to a strong heat, in a small wind-furnace, during about one hour and an half. When the crucible was broken, the oxide was found in a pulverulent state; and, from white, was become perfectly black.

In order to form a phosphuret, some phosphoric acid was poured upon a portion of the white oxide; and, being evaporated

to dryness, the whole was put into a crucible coated with charcoal, as above described. The crucible was then placed in a forge belonging to Mr. CHENEVIX ; and a strong heat was kept up for half an hour.

The inclosed matter was spongy, and of a dark brown ; it in some measure resembled phosphuret of titanium.

After this, we wished to try the effect of a still greater heat ; but in this experiment the crucible was melted.

The above experiments shew, that the white oxide, like several other metallic substances, may be deoxidated to a certain degree, without much difficulty, but that the complete reduction of it is still far from being easily effected.